

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: **Michael Krebs**

Confirmation No.: **3428**

Serial No.: **10/755,702**

Group Art Unit: **1796**

Filing Date: **January 12, 2004**

Examiner: **Patrick Dennis Niland**

For: **Reactive Polyurethanes Having Reduced Diisocyanate Monomer Content**

Mail Stop Appeal-Brief Patents
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

APPELLANT'S BRIEF PURSUANT TO 37 C.F.R. § 41.37

This brief is being filed in support of Appellant's appeal from the rejections of claims 1, 2, 4-18, 21, 24, and 28-33 dated August 24, 2007. A Notice of Appeal was filed on December 20, 2007.

1. REAL PARTY IN INTEREST

Henkel Kommanditgesellschaft Auf Aktien (Henkel KGaA) by virtue of the assignment from the inventors recorded May 5, 2004, at Reel 014600, Frame 0233.

2. RELATED APPEALS AND INTERFERENCES

Applicants are not aware of any related appeals and interferences. See appendix entitled RELATED PROCEEDINGS APPENDIX.

3. STATUS OF CLAIMS

Claims 1, 2, 4-18, 21, 24, and 28-33 are pending and are the subject of the current appeal.

4. STATUS OF AMENDMENTS

All amendments have been entered.

5. SUMMARY OF CLAIMED SUBJECT MATTER

The claimed inventions generally relate to a process for producing a reactive polyurethane containing free isocyanate groups, and in particular, reactive polyurethanes having a low percentage content of monomeric diisocyanates which are suitable both for use as reactive one- and two-component adhesives/sealants (page 5, line 27 to page 6, line 30). In the field of the invention, there is a high demand for the development of efficient processes for reducing the content of the monomeric diisocyanates (page 4, lines 15-19).

For example, the reactive polyurethanes typically used in one-component or two-component systems include terminal isocyanate (NCO) groups, and in order to obtain NCO-terminated polyurethanes, it is common practice to react polyhydric alcohols with an excess of monomeric polyisocyanates – generally diisocyanates (page 2, lines 5-10). Unfortunately, a certain quantity of the monomeric diisocyanate used in the excess is left over after the reaction, and the presence of monomeric diisocyanate is problematic in the processing of adhesives and sealants based on reactive polyurethanes (*id.* at lines 11-14).

One problem presented by the presence of monomeric diisocyanate is that isocyanate vapors are toxic in view of their irritation and sensitizing effect (*id.* at lines 14-19). At high temperatures, such as those used in hotmelt adhesives, the widely used bicyclic diisocyanates form gaseous and aerosol-like emissions that require elaborate measures to protect people responsible for applying the product (*see* page 2, line 19 to page 3, line 7). In addition, monomeric diisocyanates are capable of “migrating” from the coating or bond into the coated or bonded materials causing further problems (*see* page 3, line 14 to page 4, line 14).

The state of the art at the time of the invention was to remove excess monomeric isocyanate from the product via complicated and expensive purification steps or to utilize a two step reaction process to produce the isocyanate-terminated reactive prepolymer (*see, e.g.*, page 4, line 15 to page 6, line 15).

As stated in independent claim 1, Applicants advance the art by setting forth a process that utilizes a one step reaction of 2,4'-MDI to achieve a low monomeric isocyanate product. In particular, the process reflected in claim 1 consists of the step of reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol, wherein

(a) the monomeric asymmetrical diisocyanate used contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4% by weight;

(b) at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol is used as the polyhydric alcohol;

(c) the ratio of isocyanate groups to hydroxyl groups is a value of 1.05:1 to 2.0:1; and

(d) the reactive polyurethane thereby obtained has a content of monomeric asymmetrical diisocyanate of at most 0.3% by weight.

(See claim 1; *see also*, e.g., page 6, line 18 to page 7, line 3; page 8, lines 10-16). The remaining independent claims also recite one-step reactions to achieve a low monomeric isocyanate product. In independent claim 21, the resulting reactive polyurethane is combined with at least one additional component selected from the group consisting of catalysts, polymeric compounds, stabilizers, adhesion-promoting additives, fillers, pigments, plasticizers, and solvents (*see*, e.g., page 16, lines 4-9; page 10, lines 23-25; and page 20, lines 24-27). In the embodiment reflected in independent claim 24, the polyhydric alcohol comprises at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol and at least one polyol having a number average molecular weight of 2,000 g/mol to 20,000 g/mol selected from the group consisting of polyesters, polyethers, polyacetals, and polycarbonates (*see*, e.g., page 10, lines 16-22). Another aspect of the invention is reflected in independent claim 28, which recites a process that further reacts at least one polyol with a reactive polyurethane of the instant invention (*see*, e.g., page 14, lines 6-12).

6. GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

The issues on appeal are:

(1) Whether claims 1-2, 4-18, 21, 24, and 28-33 would have been anticipated under 35 U.S.C. § 102(b) by WO 00/05290 ("the Bolte application"),

(2) Whether claims 1-2, 4-18, 21, 24, and 28-33 would have been obvious under 35 U.S.C. § 103(a) in view of the Bolte application.

7. ARGUMENT

Claims 1-2, 4-18, 21, 24, and 28-33 Would Not Have Been Anticipated By the Bolte Application.

A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference. *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631 (Fed. Cir. 1987); MPEP § 2131. The current grounds for rejection of the claims can not stand because the Bolte application fails to disclose each and every limitation of the independent claims and therefore there can be no anticipation of the claims under 35 U.S.C. §102(b).

The Bolte application, for example, does not disclose the use of monomeric asymmetrical diisocyanate that contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4% by weight. For at least this reason, the rejection should be withdrawn.

Furthermore, claim 1 uses the transition phrase "consisting of". The transitional phrase "consisting of" excludes any element, step, or ingredient not specified in the claim. MPEP § 2111.03; *In re Gray*, 53 F.2d 520, 11 USPQ 255 (CCPA 1931); *Ex parte Davis*, 80 USPQ 448, 450 (Bd. App. 1948). As will be explained further in connection with obviousness rejection, the Bolte application teaches a two step reaction to achieve a low monomeric isocyanate product (*see*, for example, Bolte application at page 5, lines 24-30). The second stage of the process taught by the Bolte application utilizes a different difunctional diisocyanate than is used in the first stage (*see*, for example, Bolte application at page 8, line 26 to page 9, line 30; *see also* the description at page 5, line 20 to page 6, line 8 which describes reacting a polyurethane polymer (made in a first stage) with additional isocyanate components (a second stage)). The process of instant claim 1, in contrast, is a one step process. In other words, the Bolte application teaches that two steps are required to obtain a suitable low monomeric isocyanate product whereas the instant claims provide a one step process to achieve this objective. For at least this reason, the rejection should be withdrawn.

The processes claimed in independent claims 21, 24 and 28, as well as the claims that depend from these claims and claim 1 should be withdrawn for reasons analogous to those discussed for claim 1.

Claims 1-2, 4-18, 21, 24, and 28-33 Would Not Have Been Obvious in View of the Bolte Application.

To establish a *prima facie* case of obviousness, there must be some reason, either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to modify the references or to combine reference teachings. *KSR International Co. v. Teleflex Inc.*, 127 S. Ct. 1727, 1741 (2007). Moreover, the prior art reference (or references when combined) must teach or suggest all the claim limitations. The reason to make the claimed combination, and a reasonable expectation of success, must be found elsewhere than in Applicants disclosure, such as in the prior art, the nature of the problem to be solved, or in the knowledge/understanding of the person of ordinary skill in the art. MPEP § 2143; *In re Vaeck*, 947 F.2d 488, 20 USPQ2d 1438 (Fed. Cir. 1991). The rejection put forth by the Examiner does not meet these requirements.

There is no reason or motivation to modify the teachings of the Bolte application to arrive at any pending claims. No motivation is found, for example, to modify the teachings of the Bolte application to produce a one step reaction of 2,4'-MDI to achieve a low monomeric isocyanate product as recited in instant claim 1 rather than the two step process taught by the Bolte application. Indeed, the Bolte application teaches a two stage process that uses a different difunctional diisocyanate in the second stage than is used in the first stage (*see*, for example, Bolte application at page 8, line 26 to page 9, line 30). The present claims, in contrast, use a single isocyanate, 2,4'-MDI, which has two isocyanate groups of different reactivity in the same molecule. In particular, the instant claims utilize 2,4'-MDI with low levels of symmetric diisocyanate impurities. *See*, claim 1.

Although the Examiner acknowledges the use of the closed term “consisting of” in Applicants’ claims, which indicates that the reactive polyurethane containing free isocyanate groups is the result of a one-step process of reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol, the Examiner states that “it is not seen that [the Bolte application] requires two steps” (see Final Office Action at page 8-9). In particular, the Examiner argues that the passage at page 6, lines 17-21 of the Bolte application includes a teaching of a one stage reaction analogous to that of the instant claims (Final Office Action at page 9). The Examiner also states that the Bolte application does not require two different diisocyanates, but only different isocyanate groups (see Final Office Action at page 9). Applicants respectfully disagree with these assertions.

The Bolte application makes it clear that its reactive polyurethane is produced by at least a two-stage reaction. Specifically, the process of making Component A in the Bolte application is what is alleged to anticipate or render obvious the instant claims. The Bolte application identifies Component A as a polyurethane polymer containing at least two isocyanate groups or a mixture of two or more polyurethane prepolymers containing at least two isocyanate groups (*see* Bolte application at page 5, lines 24-26). The relevant question concerns the process being used to make Component A – in this respect, Bolte teaches (a) a two stage reaction with two different isocyanate compounds, or (b) mixing two prepolymers, each having one difunctional isocyanate compound (*see* page 5, line 20 to page 9, line 30). In either case, a two-step process is disclosed.

In the case of forming a polyurethane prepolymer containing at least two isocyanate groups where the two isocyanate groups are different such that one has a lower reactivity to isocyanate reactive groups than the other, the Examiner asserts that the Bolte application does not require two different diisocyanates in the process, but only different isocyanate groups (*see* Final Office Action at page 9). Such an assertion is belied by the Bolte disclosure.

For example, to support the assertion, the Action cites page 7, line 23-30 and page 8, lines 1-2 of the Bolte application (Final Office Action at page 9-10). These passages, however, state that such a prepolymer is obtained from the reaction of a dihydric alcohol with **compounds** containing two different difunctional, isocyanate groups such that each molecule of the dihydric alcohol reacts with one molecule of the **compounds** containing different isocyanate groups (*see* Bolte application at page 7, line 23 to page 8, line 2, emphasis added). The Bolte application uses the plural “compounds” to evidence the more than one compound having different isocyanate groups involved in the process, not a single isocyanate which has two isocyanate groups of different reactivity in the same molecule as in the instant claims.

This interpretation of the Bolte application is fully supported by clear explanations of how such an embodiment is actually carried out, namely by a two-stage reaction in which (1) in a first stage, a polyurethane prepolymer is prepared from at least difunctional isocyanate and at least a first polyol component and (2) in a second stage, another at least difunctional isocyanate is reacted with the polyurethane prepolymer from the first stage, wherein the isocyanate groups of the isocyanate added in the second stage have a higher reactivity to isocyanate reactive compounds than at least the predominant percentage of the isocyanate groups present in the polyurethane prepolymer from the first stage (*see* Bolte Application at page 8, line 29 to page 9, line 30). Taking the disclosure as a whole supports Applicants’

argument that the Bolte application discloses two-stage reactions to produce Component A. Descriptions of the individual isocyanates that have different reactive groups (*see* Bolte application at page 15, lines 15-25) that may be used in the process does not change the fact that Bolte teaches two-stage processes to arrive at its Component A.

Additional support for the fact that the Bolte application is teaching two-stage processes concerns the Bolte application's requirement that its end product (Component A) contain two different reactive NCO-groups, either as a mixture of polymers or in one polymer. To the extent a single isocyanate with two different reactive groups, one group having higher reactivity than the other, is being used in a one-stage reaction, the faster group will react first and the slower group thereafter, resulting in only the slower group remaining after the completion of the reaction. Such a result would be inconsistent with Component A containing two different reactive groups. Not only does this indicate that the process in Bolte is not a single-stage reaction, it cuts against modifying the process of the Bolte application to be a single stage application. It also highlights the difference in the end-product resulting from the process of the instant claims and that resulting from the process of the Bolte application.

Furthermore, it would not be obvious from the Bolte application, that a one stage process would be capable of producing a reactive polyurethane with a free monomeric isocyanate content of not more than 0.3%. The state of the art, as discussed in the Discussion of the Related Art of the application (page 1, line 13 to page 6, line 15), was to remove excess monomeric isocyanate from the product or to utilize a two step reaction process to produce the isocyanate-terminated reactive prepolymer. The Bolte application does not teach or suggest that a low free monomeric isocyanate content would be possible by the one step method of the instant claims. Rather, the Bolte application, as is consistent with the other art, teaches that a two stage approach is appropriate to lower the free isocyanate content of a product containing isocyanate groups of different reactivity. As such, the claimed one step process is not obvious in view of the Bolte application.

For at least these reasons, the instant claims are not obvious in view of the teachings of the Bolte application. Withdrawal of the rejection is respectfully requested.

The rejections of independent claims 21, 24 and 28, as well as the claims that depend from these claims and claim 1 should be withdrawn for reasons analogous to those discussed for claim 1.

8. CONCLUSION

For the foregoing reasons, it is respectfully submitted that the Patent Office has not met its burden of establishing that claims 1-2, 4-18, 21, 24, and 28-33 are anticipated under 35 U.S.C. § 102(b) or obvious under 35 U.S.C. § 103(a). Reversal of the final rejections and the issuance of a notice of allowance for the appealed claims are respectfully requested.

Respectfully submitted,

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9. CLAIMS APPENDIX

1. A process for producing a reactive polyurethane containing free isocyanate groups, said process consisting of the step of reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol, wherein:

- (a) the monomeric asymmetrical diisocyanate used contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4% by weight;
- (b) at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol is used as the polyhydric alcohol;
- (c) the ratio of isocyanate groups to hydroxyl groups is a value of 1.05:1 to 2.0:1; and
- (d) the reactive polyurethane thereby obtained has a content of monomeric asymmetrical diisocyanate of at most 0.3% by weight.

2. A process as claimed in claim 1, wherein the reactive polyurethane has a Brookfield viscosity at 100°C, as measured by ISO 2555, in the range from 20 mPas to 3,000 mPas.

4. A process as claimed in claim 1, wherein at least one linear or lightly branched C₂₋₁₈ alkanediol is used as the diol.

5. A process as claimed in claim 1, wherein said reacting is carried out in the presence of at least one catalyst selected from the group consisting of organometallic compounds of tin, lead, iron, titanium, bismuth and zirconium.

6. A process as claimed in claim 1, wherein said reacting is carried out between 30°C and 130°C in the presence of a tin(IV) compound as catalyst.

7. A process as claimed in claim 1, wherein said reacting is carried out at 40 to 75°C.

8. A process as claimed in claim 1, wherein the ratio of isocyanate groups to hydroxyl groups is adjusted to a value of 1.05:1 to 1.5:1.

9. A process as claimed in claim 1, wherein said reacting is carried out between 25°C and 100°C.

10. A process as claimed in claim 1, wherein said at least one diol contains secondary hydroxy groups.

11. A process as claimed in claim 1, wherein said reacting is carried out in the presence of an aprotic solvent.

12. A process as claimed in claim 1, wherein said reactive polyurethane contains not more than 0.1% by weight monomeric asymmetrical diisocyanate.

13. A process as claimed in claim 1, wherein the reactive polyurethane has an NCO content of from 4.5 to 10% NCO.

14. A process as claimed in claim 1, wherein said at least one diol has a number average molecular weight of 200 g/mol to 1,500 g/mol.

15. A process as claimed in claim 1, wherein said at least one diol is a polyether.

16. A process as claimed in claim 1, wherein said at least one diol is selected from the group consisting of reaction products of low molecular weight polyhydric alcohols and alkylene oxides containing 2 to 4 carbon atoms.

17. A process as claimed in claim 1, wherein said at least one diol is polypropylene glycol.

18. A process as claimed in claim 1, wherein the ratio of isocyanate groups to hydroxyl groups is adjusted to a value of 1.4:1 to 1.9:1.

21. A process for producing a reactive polyurethane containing free isocyanate groups, said process consisting of:

- (i) reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol,

wherein:

- (a) the monomeric asymmetrical diisocyanate used contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4;
 - (b) at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol is used as the polyhydric alcohol;
 - (c) the ratio of isocyanate groups to hydroxyl groups is a value of 1.05:1 to 2.0:1;
 - (d) the reactive polyurethane thereby obtained has a content of monomeric asymmetrical diisocyanate of at most 0.3% by weight; and
- (ii) combining the product of step (i) with at least one additional component selected from the group consisting of catalysts, polymeric compounds, stabilizers, adhesion-promoting additives, fillers, pigments, plasticizers, and solvents.

24. A process for producing a reactive polyurethane containing free isocyanate groups, said process consisting of the step of reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol, wherein:

- (a) the monomeric asymmetrical diisocyanate used contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4;
- (b) the polyhydric alcohol comprising at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol and at least one polyol having a number average molecular weight of 2,000 g/mol to 20,000 g/mol selected from the group consisting of polyesters, polyethers, polyacetals, and polycarbonates; wherein the ratio of isocyanate groups to hydroxyl groups is a value of 1.05:1 to 2.0; and
- (c) the reactive polyurethane thereby obtained has a content of monomeric asymmetrical diisocyanate of at most 0.3% by weight.

28. A process for producing an isocyanate-terminated reactive polyurethane composition, said process comprising reacting at least one polyol with a reactive polyurethane containing free isocyanate groups made by a process consisting of the step of reacting a monomeric asymmetrical diisocyanate with a polyhydric alcohol, wherein:

- (a) the monomeric asymmetrical diisocyanate used contains at least 95% by weight of diphenylmethane-2,4'-diisocyanate (2,4'-MDI) and less than 5% by weight of 4,4'-MDI and 2,2'-MDI, the 2,2'-MDI content being under 0.4;
- (b) at least one diol with a number average molecular weight of 60 g/mol to 2,000 g/mol is used as the polyhydric alcohol;
- (c) the ratio of isocyanate groups to hydroxyl groups is a value of 1.05:1 to 2.0:1; and
- (d) the reactive polyurethane thereby obtained has a content of monomeric asymmetrical diisocyanate of at most 0.3% by weight.

29. The process of claim 28, wherein an NCO:OH ratio of up to 10 is used.

30. The process of claim 28, wherein an NCO:OH ratio of from 1.2:1 to 5:1 is used.

31. The process of claim 28, wherein said at least one polyol is a polyester polyol.

32. The process of claim 1, wherein the reactive polyurethane has an NCO content of from 4 to 12% NCO.

33. The process of claim 1, wherein the reactive polyurethane has an NCO content of from 5 to 8% NCO.

10. EVIDENCE APPENDIX

There is no additional evidence provided.

11. RELATED PROCEEDINGS APPENDIX

None.